Fabrication of Ultra-Thin Suspended ALD Membranes

Michael Elowson, Rohan Dhall, Adam Schwartzberg, Stephanie Chang, Vittoria Tommasini, Sardar Alam, Stefano Cabrini, Shaul Aloni
The Molecular Foundry, Lawrence Berkeley National Laboratory, Berkeley, CA 94720
melowson@lbl.gov

Ultra-thin films play a major role in micro- and nano-fabrication, where their electronic, optical, and chemical properties allow for functional interfaces such as electrically insulating layers, anti-reflective coatings, and protective masks[1]. Many electron and photon characterization techniques benefit from ultra-thin sample support structures, or “windows,” which interact as little as possible with the incident beams. In transmission electron microscopy (TEM), a thinner window composed of elements with lower atomic numbers will result in less inelastic electron scattering, thus improving the contrast and resolution of the TEM image[2]. However, the number of window materials available with standard and scalable fabrication techniques remains very limited to this day[3].

Here we demonstrate a novel fabrication process for creating ultra-thin suspended films from a variety of materials, for preliminary use as TEM windows. The process involves fabricating Si_3N_4 windows with commonly-used methods, followed by deposition of a polymer etch stop and subsequently an ultra-thin ALD film, which is later released as a free-standing membrane by a backside plasma etch (Figure 1). The high chemical selectivity between ALD films and the polymer etch stop means that ultra-thin films of a variety of materials can be released without damage using a low-power O_2 plasma. ALD films as thin as 5 nm have been successfully released with lateral dimensions of 20 µm x 20 µm.

TEM was conducted on 10 nm-thick titanium nitride (TiN) membranes to characterize the ALD depositions as well as evaluate their usefulness as TEM windows. Gold nanoparticles were imaged, with high contrast observed between the particles and the window material (Figure 2b). Electron energy loss spectroscopy (EELS) spectra were taken to measure the inelastic electron mean free path (Figure 2d), as well as to characterize the chemical composition of the TiN window (Figure 2e).

Ultra-thin windows can improve contrast and resolution in TEM due to reduced scattering. Additionally, the flexibility and precision of ALD allow for fabrication of sample support structures with finely tunable chemical, structural, and optical properties. Functional materials such as conductive TiN can reduce beam charging as well as be integrated into experiments as a biasing electrode, electroplating base, or catalyst for in-situ experiments. Use of the membranes to seal liquids for in-situ liquid TEM experiments is also being developed.

The work at the Molecular Foundry was supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. DOE under Contract No. DE-AC02-05CH11231. We would like to acknowledge Hilary Brunner and Dominik Ziegler of Scuba Probe Technologies for the use of their Parylene-C deposition system.

---

[1] L. Reimer, *Transmission Electron Microscopy: Physics of Image Formation and Microanalysis*. Springer, 2013.
[2] A.S.H. Makhlouf and I. Tiginyanu, *Nanocoatings and Ultra-Thin Films Technologies and Applications*. Woodhead Publishing Limited, 2011.
[3] J.R. Dwyer and M. Harb, *Through a Window, Brightly: A Review of Selected Nanofabricated Thin-Film Platforms for Spectroscopy, Imaging, and Detection*, Applied Spectroscopy. 2017;71(9):2051-2075.

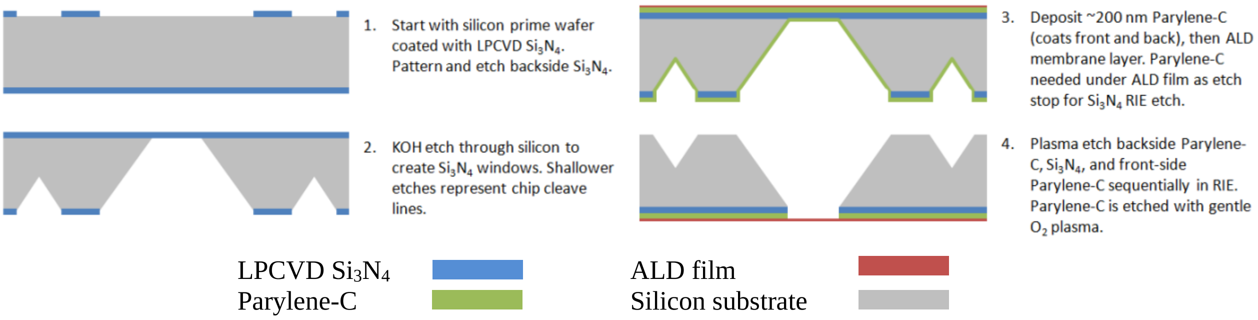

Figure 1: Novel process for fabrication of ultra-thin suspended ALD membranes. The process is agnostic to the top ALD film, which becomes the suspended membrane, as long as it is deposited below 150 C and is not damaged by low-power O_2 plasma.

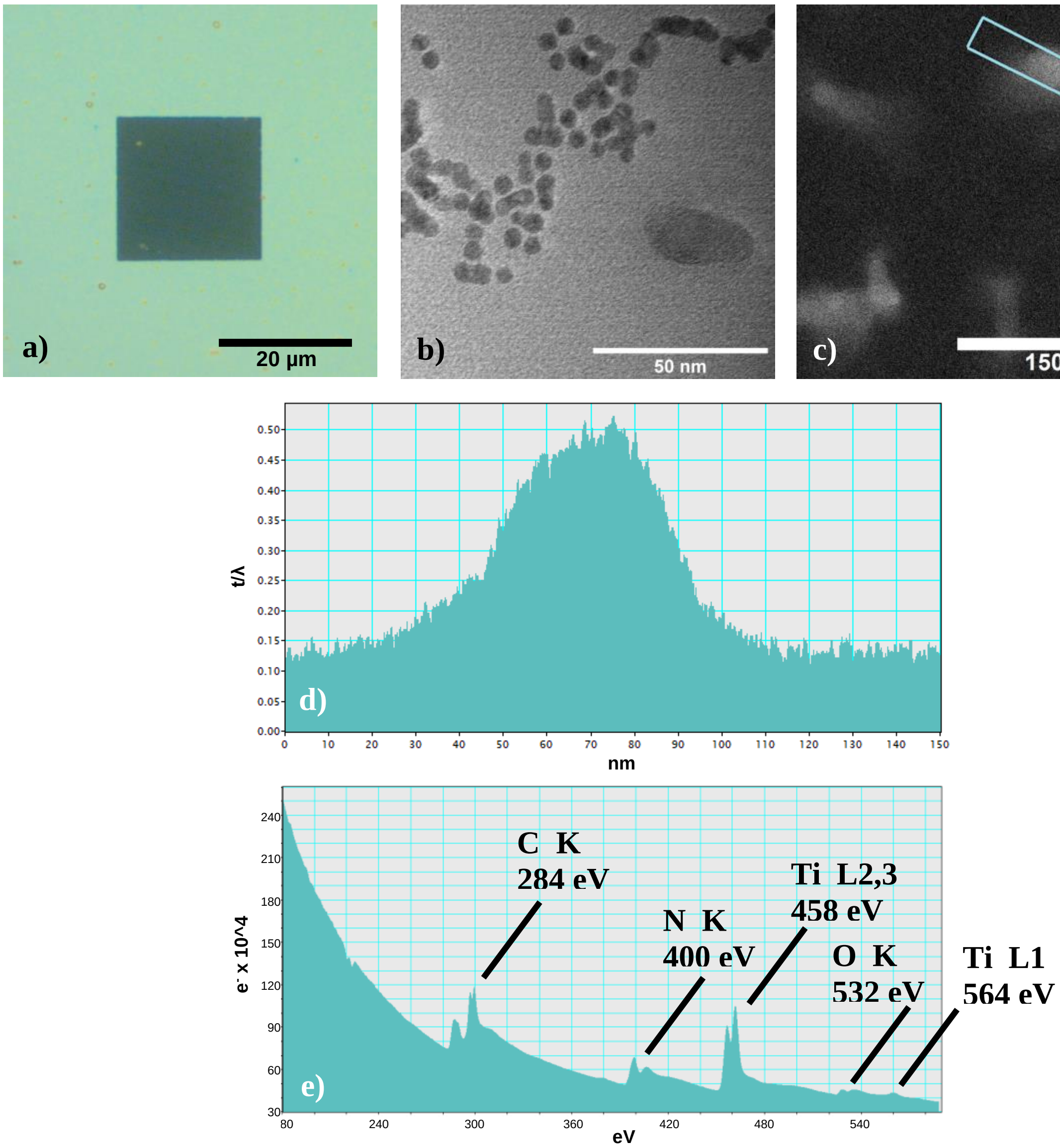

Figure 2: (a) 20 µm x 20 µm suspended membrane of 10 nm-thick conductive TiN deposited using plasma ALD at 40 C. (b) TEM image of gold nanoparticles on a 10 nm-thick conductive TiN membrane deposited using plasma ALD at 100 C. (c) Thickness map showing carbon contamination on 10 nm-thick TiN membrane. (d) Line profile of rectangular area highlighted in (c), indicating sample and contamination thickness in units of t/λ (thickness divided by inelastic mean free path). (e) EELS core loss spectra of suspended TiN membrane, indicating some carbon and oxygen contamination.